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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.039
 wR factor = 0.097
Data-to-parameter ratio = 14.8

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

(2,4-Dichlorobenzoylmethyl)triphenylphosphonium chloride

In the crystal structure of the title compound, $\text{C}_{26}\text{H}_{20}\text{Cl}_2\text{OP}^+\cdot\text{Cl}^-$, there are intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, and both intramolecular and intermolecular $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

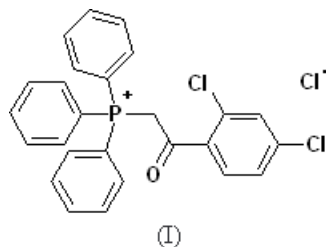
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Comment

Triphenylphosphonium salts are often intermediates in the preparation of triphenylphosphonium ylides, which are important materials in Wittig reactions (Maryanoff & Reitz, 1989). Triphenylphosphonium salts can also react with aldehydes in the absence of base to form a $\text{C}=\text{C}$ double bond (Zhang *et al.*, 2004). Some crystal structures of these salts have been reported (Antipin & Struchkov, 1984). The title compound, (I), is an intermediate in the preparation of 1-phenylpropenones.



In the molecule of (I) (Fig. 1), the planarity of the substituted phenyl ring C21–C26 (r.m.s. deviation = 0.007 Å) is unaffected by the chloro substituents. There are standard electrostatic interactions between the (2,4-dichlorobenzoylmethyl)triphenylphosphonium cations and the chloride anions.

As in other triphenylphosphonium salts (Mariyatra *et al.*, 2003), there are some intermolecular and intramolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds (Table 2). The packing is shown in Fig. 2.

Experimental

The title compound was synthesized from triphenylphosphine (2.62 g, 10 mmol) and 2,2',4'-trichloroacetophenone (2.23 g, 10 mmol), refluxed for 2 h in toluene (50 ml). The product was separated by filtration. Single crystals suitable for X-ray diffraction (m.p. 423–425 K) were obtained by slow evaporation of a benzene/toluene (1:2) solution. Spectroscopic analysis: IR (KBr, ν cm^{-1}): 2997, 2782, 1659, 1107; ^1H NMR (CDCl_3 , δ): 7.42–7.95 (*m*, 18H), 6.62 (*s*, 2H).

Crystal data

$C_{26}H_{20}Cl_2OP^+ \cdot Cl^-$
 $M_r = 485.74$
 Monoclinic, $P2_1/c$
 $a = 12.5615$ (4) Å
 $b = 10.9659$ (4) Å
 $c = 17.2638$ (5) Å
 $\beta = 104.805$ (1)°
 $V = 2299.11$ (13) Å³
 $Z = 4$

$D_x = 1.403$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4946 reflections
 $\theta = 2.5$ – 25.2 °
 $\mu = 0.49$ mm⁻¹
 $T = 298$ (2) K
 Block, colorless
 $0.26 \times 0.21 \times 0.19$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{min} = 0.884$, $T_{max} = 0.914$
 11818 measured reflections

4131 independent reflections
 3694 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.021$
 $\theta_{max} = 25.2$ °
 $h = -15 \rightarrow 13$
 $k = -13 \rightarrow 13$
 $l = -20 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.097$
 $S = 1.06$
 4131 reflections
 280 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.8375P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.34$ e Å⁻³
 $\Delta\rho_{min} = -0.27$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C11–C22	1.737 (2)	P1–C19	1.8044 (19)
C12–C24	1.733 (2)	O1–C20	1.218 (2)
P1–C6	1.7984 (19)	C19–C20	1.526 (3)
P1–C18	1.8000 (19)	C20–C21	1.479 (3)
P1–C12	1.8006 (18)		
C6–P1–C18	103.99 (9)	O1–C20–C21	123.30 (17)
C6–P1–C12	107.65 (9)	O1–C20–C19	117.84 (17)
C18–P1–C12	113.76 (9)	C23–C22–C11	115.44 (15)
C6–P1–C19	111.49 (9)	C21–C22–C11	122.91 (16)
C18–P1–C19	111.28 (9)	C25–C24–C12	119.12 (17)
C12–P1–C19	108.59 (9)	C23–C24–C12	118.55 (16)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C3–H3 ⁱ ···Cl3 ⁱ	0.93	2.82	3.640 (2)	148
C9–H9 ⁱⁱ ···O1 ⁱⁱ	0.93	2.51	3.386 (3)	157
C17–H17 ⁱⁱⁱ ···Cl2 ⁱⁱⁱ	0.93	2.79	3.396 (2)	123
C19–H19A ^{iv} ···Cl3 ^{iv}	0.97	2.59	3.534 (2)	165
C19–H19B ^v ···Cl3	0.97	2.59	3.555 (2)	173
C23–H23 ^v ···Cl3 ^v	0.93	2.80	3.715 (2)	166

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $1 - x, -y, 1 - z$; (iv) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (v) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

All H atoms were initially located in a difference Fourier map and were then positioned geometrically and constrained to ride on their parent atoms, with C–H distances in the range 0.95–1.0 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXTL.

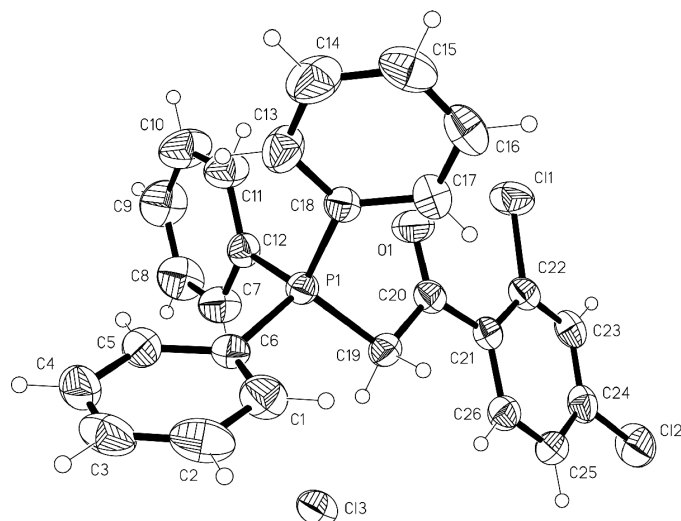


Figure 1

The structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

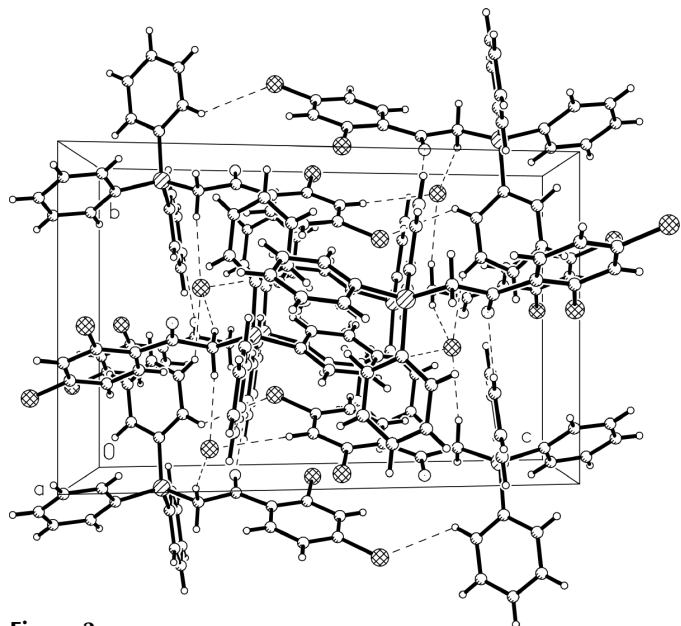


Figure 2

Packing diagram, viewed down the a axis. Dashed lines represent hydrogen bonds.

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References

- Antipin, M. Y. & Struchkov, Y. T. (1984). *J. Struct. Chem.* **25**, 122–122.
 Bruker (2002). SMART, SAINT, SADABS and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
 Mariyatra, M. B., Kalyanasundari, B., Panchanatheswaran, K. & Goeta, A. E. (2003). *Acta Cryst.* **E59**, o255–o257.
 Maryanoff, B. E. & Reitz, A. B. (1989). *Chem. Rev.* **89**, 863–927.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Zhang, X., Zhong, P. & Chen, F. (2004). *Synth. Commun.* **34**, 1729–1736.